Patented Aug. 29, 1933 1,924,890

UNITED STATES PATENT OFFICE

1,924,890

METHOD OF PREPARING GELATIN BLANKS

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No Drawing. Application June 12, 1929
Serial No. 370,470

15 Claims. (Cl. 91—70)

This invention relates to a method of hardening gelatin compositions and to the resulting product or products obtained by the application of the method. It finds its most typical exemplification in preparing the gelatin coatings upon transparent films such as the cellulosic films commonly used in photography and for motion picture reproductions.

It has been proposed in copending application (Serial No. 105,876, filed April 30, 1926, now United States Patent 1,865,497, issued July 5, 1932) to treat gelatin compositions with a hardening agent and with an organic acid, and thereafter to effect the hardening of the gelatin by subjecting the same to heating.

The present invention includes or assumes a preliminary preparation of a solution or composition containing gelatin (which may contain an organic acid or a hardening agent) which is extended to form the surface desired, either as a gelatin film or as a surface coating, and preferably while in the fluid condition or in the form of a partially set surface or sheet and before complete drying, then adding the hardening agent and/or organic acid, in solution or gaseous condition, followed by effecting or completing the set of the gelatin. This is preferably carried to the obtainment of a firm but moist consistency, as by drying (preferably promptly and at low temperature) and hardening by subjecting to wave radiation such as light and/or heat, preferably the latter.

In the present disclosure, the hardening agent is understood to be a reagent which per se is not effective to harden the gelatin like chrome alum but which, in the presence of a third reagent such as an organic acid, initiates the hardening action and carries it through to completion; especially with appropriate application of light or heat.

For example, a coating composition of gelatin of suitable concentration and containing an appropriate hardening agent (e. g. 5% potassium bichromate) may be applied to a backing layer such as transparent celluloid film or the like. The coating thus obtained is then allowed to partially dry and set. Before complete setting, however, the coated film is subjected to the action of an activating acid such as citric or acetic acid (e. g. 10% solution) by passing the same through a solution or bringing into contact with the acid in gaseous condition. Thereupon the gelatin coating adsorbs or dissolves a relatively large proportion of the acid, in the order of 5%–20% if calculated upon the weight of the gelatin. The film is then dried to a firm consistency, whereupon a part of the hardening action of the gelatin may take place. It is then subjected to an elevated temperature (e. g. 90–110° F.) which completes the hardening of the film and substantially or completely expels any last traces of the acid (if volatile) so that subsequent continued hardening of the gelatin with age is prevented.

In another aspect of the invention the transparent backing film of celluloid or like material may be first treated with a dilute solution of the organic acid (e. g. 10% acetic acid and preferably with alcohol as the solvent) and a small amount of gelatin. The film is then allowed to dry slightly, enough to preserve uniformity of distribution of the applied coating but without substantial evaporation or loss of the adsorbed (or absorbed) acetic acid.

The gelatin coating comprising a solution or emulsion of gelatin may now be prepared with water as the solvent and in such relative proportions as may be necessary to produce the desired consistency (e. g. 10%) and approximately 5% of potassium bichromate upon the weight of the gelatin is added as the hardening agent.

The acid treated film is then provided with a layer of the coating composition as by dipping, and allowed to dry or set to a sufficiently firm consistency to permit convenient handling of the coated film. The film is then dried further if necessary and subjected to an elevated temperature, preferably between 90° and 110° F., for a sufficient time to effect the desired degree of hardening. The acetic acid applied to the backing more or less gradually permeates the gelatin and hardening agent and accelerates the hardening action thereof. It is usually desirable so to effect the drying and hardening operations that the free organic acid, if volatile as when acetic acid is used, shall be substantially completely expelled and further hardening inhibited.

This aspect of the invention is substantially the aquivalent of the procedure above described in which the gelatin layer first applied to the backing contains a hardening agent and the second gelatin coating contains the acid, and such transposition of steps is also to be understood as comprehended by the claims.

It is thought that the acid (specifically acetic or citric acid in the instant case) facilitates or accelerates the hardening action of the bichromate upon the gelatin coating. In any event, such acceleration is more especially effective in

fluid or water-moist media, but is appreciably retarded or prevented altogether when the gelatin coating becomes thoroughly dry. Accordingly, the introduction of either the acetic acid or the bichromate may be delayed until the gelatin coating containing one of the reagents has acquired a firm consistency, or partially set, before the other is added as above described, but not until it has become hard and dry. Hardening may then be effected by a rise of temperature (causing the acid to permeate the moist coating layer from the backing) or the hardening action may be activated by the acid, if the latter is applied subsequently in solution or in the form of a gaseous medium such as acetic acid vapors. In either case, upon permeating the gelatin coating (whether partially set or of fairly firm consistency) the reagents become effective to initiate and facilitate the hardening reaction. The hardening action is further promoted by rise of temperature and the degree of hardening may be regulated by the relative proportions and concentrations of the reagent materials present, the time and temperature of treatment, and the atmospheric conditions surrounding the same, the combined effect determining the degree and rate at which the organic acid is expelled in the event that the organic acid is volatile. The acid on the backing may be more fully driven into and through the gelatin coating by continuing the heating and any excess of free acid may be substantially completely expelled from the film, by prolonging the heat treatment.

The gelatin film as thus produced is characteristically acidic, whether a non-volatile or volatile organic acid has been employed. In the latter case, the acid may be in greater part removed by the heating and evaporation as just pointed out and there may in fact be no free acid left in or on the gelatin. But in such case the normally amphoteric gelatin is nevertheless left in an acidic condition. A quality attributable to such acidic condition of the films is found to be that images in certain acid dyes such as red S are more accurately transferred and reproduced thereon (as by imbibition printing) than when the gelatin surface is in other than an acid condition and printed with a dye solution which has been acidified according to the more or less common practices of the art.

In the latter circumstances, it is found that not only is the dye less accurately transferred physically but that also some reaction effect is produced upon the color substance of the dye whereby the color value and density is changed so that the various shades are not true and are not governed by the relative densities of their distribution alone but are affected and offset, giving other and uncontrollable effects in the final product.

It is further found that, in accordance with this invention, the presence of an organic acid which may be absorbed (or perhaps in chemical combination) throughout the gelatin mass to a concentration equivalent to 5 to 20% upon the weight of the gelatin is especially effective to insure obtaining a suitable film for printing with acid dyes such as fast red S without such change or loss of color value of the dye.

I claim:

1. A process of treating gelatin compositions which comprises the steps of treating the same in moist, partially set condition, containing an acid-activated hardening agent, with a solution containing gelatin and an activating organic acid, and causing the hardening reaction to proceed.

2. A process of hardening gelatin, which comprises the steps of treating the same in partially set condition and containing an acid-activated hardening agent, with a gelatin solution containing an activating organic acid, and thereafter causing the hardening reaction to proceed and the gelatin to completely set.

3. A process of hardening gelatin, which comprises the steps of treating the same in partially set condition and containing an acid-activated hardening agent, with a gelatin solution containing an activating organic acid, and subjecting to radiation.

4. A process of hardening gelatin, which comprises the steps of treating the same in partially set condition and containing an acid activated hardening agent, with a gelatin solution containing an activating organic acid, and subjecting to light.

5. A process of hardening gelatin, which comprises the steps of treating the same in partially set condition and containing an acid activated hardening agent, with a gelatin solution containing an activating organic acid, and subjecting to an elevated temperature.

6. A process of hardening gelatin, which comprises the steps of treating the same in partially set condition and containing an acid activated hardening agent, with a gelatin solution containing an excess of an activating organic acid, and subjecting to an elevated temperature.

7. A process of hardening gelatin, which comprises the steps of treating the same in partially set condition and containing an acid activated hardening agent, with a gelatin solution containing an excess of an activating volatile organic acid, and subjecting to an elevated temperature.

8. A process of preparing hardened gelatin coatings, which comprises the steps of extending a gelatin solution or emulsion in a relatively fluid condition and containing an acid-activated hardening agent to form a film or coating, partially setting the mass to a firm consistency, treating with a solution of gelatin and an activating acid, and thereafter subjecting the treated composition to radiation, to harden the same.

9. A process of preparing hardened gelatin coatings, which comprises the steps of incorporating an acid activated hardening agent with gelatin in a relatively fluid condition, extending the same to form a film or coating, partially setting the mass to a moist, firm consistency, treating with a solution of gelatin and a volatile activating acid, and subjecting the treated composition to an elevated temperature.

10. A process of preparing hardened gelatin coatings, which comprises applying to a backing film or surface a liquid composition containing gelatin, and an acid activated hardening agent, treating with a solution containing gelatin and an activating acid, and thereafter subjecting the coated film to radiation.

11. A process of preparing hardened gelatin coatings, which comprises applying to a backing film a liquid composition containing gelatin and an acid activated hardening agent, treating with a solution containing gelatin and a volatile organic acid, and subjecting the thus coated film to an elevated temperature.

12. A process of preparing hardened gelatin coatings, which comprises applying to a backing film a liquid composition containing gelatin and an acid activated hardening agent, treating with a solution containing acetic acid and gelatin, and

subjecting the coated film to an elevated temperature.

13. A process of preparing hardened gelatin-coated films, which comprises applying to a transparent film a liquid coating of gelatin and a bichromate an acid activated hardening agent, setting the gelatin coating to a firm consistency, treating the same with a solution containing gelatin and an excess of a volatile organic acid, and thereafter subjecting the same to an elevated temperature.

14. A process of preparing hardened gelatin-coated films, which comprises applying to a transparent film a liquid coating of gelatin and an acid activated hardening agent, treating the same with a solution containing gelatin and excess of acetic acid, and thereafter subjecting the same to an elevated temperature.

15. A film or surface coating characterized by comprising a layer of gelatin containing an acid activated hardening agent and a gelatin layer containing an excess of an acid, said layers being integrally joined and hardened by the interpenetration of the hardening agent and acid.

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